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(71)Applicant : NIPPON KOUYU:KK

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(72)Inventor : TAKEMURA KUNIO
SAITO TAKASHI

(54) PRODUCTION OF LOW-NOISE UREA GREASE COMPOSITION

(57)Abstract:

PURPOSE: To obtain the subject composition having improved heat-resistance and durability by heating a mixture of a specific urea compound and a base oil to completely dissolve the components and cooling the mixture at a definite rate of cooling.

CONSTITUTION: The objective composition can be produced by mixing (A) 2-30wt.% of a urea compound of formula (R1 and R3 are 8-18C saturated alkyl; R2 is tolylene, diphenylmethane or dimethyl-biphenylene) and (B) 98-70wt.% of a base oil comprising a mineral oil, a silicone oil, a phosphate oil, etc., heating the mixture at 170-230°C to completely dissolve the urea compound into the base oil and cooling the mixture at a cooling rate of $\geq 5^\circ\text{C}/\text{sec}$ (preferably $\geq 10^\circ\text{C}/\text{sec}$).

~~R1 - NHCONH - R2 - NHCONH - R3~~

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[WHAT IS CLAIMED IS:]

[Claim 1]

Nitramine, R1-NHCONH-R2-NHCONH-R3

[, out of equation, R2 expresses tolylene radical, diphenyl-methane radical or dimethyl biphenylene radical in a saturation alkyl group of carbon number 8-18 R1 and R3.]

Formula of low noise urea grease composition including grease having high heat resistance being provided than a case of one kind of grease because it is from tertiary operation mixing higher than two kinds of grease comprising of foreign urea chemical agent among grease provided by the second operation making cool mixture comprising of 2-30 urea compound % by weight and 98-70 base oil % by weight which it appears, and is jisa to room temperature at the rate of higher than 5 degrees Celsius per second after the first operation making it heats, and 170-200 degrees Celsius dissolve urea chemical agent in complete and the first operation and the second said operation.

[DETAILED DESCRIPTION OF THE INVENTION]

[a field of industrial application] the present invention relates to formula of urea grease composition used for bearing called for low noise.

[prior art: and the problems], besides, abatement of harsh noise becomes a problem along with function improvement of the original in motor accessories home electric appliance, OA apparatus from technical development and high-quality centromere orientation of a user of late years. Bearing supporting electric motor rotation axis of rotary device as one of source of release of noise of such apparatus is given. It is it is with sound occurring from other division as for the sound to occur from this bearing, and to push up noise level of the whole equipment. For a cause of this bearing noise, processing assembling accuracy of bearing, effect of the grease medius foreign matter and zo* agent corpuscule which it is installed, and employ as issue of accuracy, lubricant are given, but, in late years it is noise due to lubricating grease that it is with issue. In other words, When foreign matter and zo* agent corpuscule in the lubricating grease which invaded a rolling element of bearing and a minute gap between *things* of *ten* race side break, and it is destroyed, it makes bearing oscillate, it appears as noise of bearing. In bearing called for low noise, lithium soap base grease produced by manufacturing process managed for debris contamination prevention closely is used broadly, but, the grease which showed the ability which was long term stable became demand from even high temperature by *ka*ka* of service condition of late years. It is compared with lithium soap base grease, and, as against such a claim, trial to use urea grease having the gel structure which is stable does even a high temperature, but, urea grease is easy to generate bearing noise in character of the zo* agent corpuscule, most do not spread in bearing called for noise low. As a result of having analyzed as concerns the urea grease that scholars of present invention marketed at first to study a cause of bearing noise development by urea grease, even if length of a particle of unit corpuscule of grease medius urea chemical agent (or unit fiber, the following) (or, fiber length) is massive, though most are less than five mu m in ten mu m dimension, there is aggregate of 20-200 mu m dimension which the unit corpuscule gathered a lot, this aggregate hit a certain thing at the bottom of bearing noise, and it was stopped. In addition, About amine shown in follows, diisocyanate, combination of base oil each, a weight ratio of urea chemical agent and base oil to generate by reaction of amine and diisocyanate makes the combination that it seems to be to 10 to 90, base oil solution of base oil solution and diisocyanate of amine is mixed, and it is reacted, heat agitation is processed to 170 degrees Celsius, as a result of it was produced experimentally by formula of the general which it was finished in three roll mill after *horei* to room temperature, and was processed, and having analyzed, aggregate of 20-200 mu m dimension ensured a mass existing thing in all trial manufacture grease. As a result that, even more particularly, amine n- propylamine, isopropylamine n-butylamine, isobutyl amine s- n-butylamine, n- pentylamine 3- methylbutyl amine ,n - heptyl amine n- octyl amine, ,2- ,4- n- tetradecylamine n- dodecylamine ethylhexyl amine n- hexadecyl amine ,n - octadecylamine aniline biphenyl amine p- phenetidine, p- anisidine p- dodecyl aniline,

cyclopentylamine cyclohexylamine, dihydro Gavia stellata ethamine, 1,5- 4,4 2,4 3,3 4,4 3,5,5-trimethyl hexylamine diisocyanate *nafuchirenjiisoshianeto* □CE - diphenyl-methane diisocyanate □CE - tolylene diisocyanate □CE - dimethyl - □CE - biphenylene diisocyanate hexamethylene diisocyanate base oil alpha - olefin oligomer (40 degrees Celsius kinematic viscosity 30cSt) paraffin series liquid petrolatum (40 degrees Celsius kinematic viscosity 67cSt) dimethyl silicone (25 degrees Celsius kinematic viscosity 200cSt) pentaerythritol tetraester (40 degrees Celsius kinematic viscosity 31cSt) examined variation of aggregate to be able to put within manufacturing process about two kinds of grease which reacted grease, cyclohexylamine and 4, four □CE - diphenyl-methane diisocyanate which reacted n- octyl amine and 4,4 □CE - diphenyl-methane diisocyanate in paraffin series liquid petrolatum (40 degrees Celsius kinematic viscosity 67cSt) in pentaerythritol tetraester (40 degrees Celsius kinematic viscosity 31cSt), there is already aggregate to two kinds as of reaction, the some increase and decrease ensured that a certain thing did not disappear with migration of operation. That about around 50% were decreased by when it passed 20 times in three roll mill to have been effective in reducing this aggregate in operation was understood. 100 times more are passed, and some abatement is accepted, but, it does not disappear in complete and such a method needs unreasonable time and effort, it is hard to be offered for practical use. Homogenizer aftertreatment, ball mill aftertreatment, colloid mill treatment were tried as other finish art, but, the prominent effect was not provided.

[measure to solve a problem] various kinds of method is devised in mechanical treatment as above because there is boundary, as a result of having tried, it makes mixture comprising of urea chemical agent of less than or equal to melting point 230 degrees Celsius and base oil is heated, and dissolve urea chemical agent in base oil, 20 μ m losing aggregate older than by subsequently cooling these 5 degrees Celsius per second solution to room temperature at the rate of the above was succeeded in. By this method, The disperse system which is homogeneity of minute urea compound crystal of less than or equal to five grain length of a particle (or, fiber length) μ m is provided, a thing having low noise ability superior to marketing lithium soap base grease became clear. Urea compound to use for the present invention is shown in the next nitramine. R1-NHCONH-R2-NHCONH-R3

[, out of equation, R2 expresses tolylene radical, diphenyl-methane radical or dimethyl biphenylene radical in a saturation alkyl group of carbon number 8-18 R1 and R3.]

Melting point goes over 230 degrees Celsius to phenyl group, Crea chemical agent including cycloalkyl group in R3 that carbon number of R1 and R3 invites urea compound of less than or equal to 7 and R1 in this equation, because it is risk and even if when it must heat larger than fire point of base oil, there is, and it heats larger than melting point, it is not suitable for the present invention so that there is when it is not dissolved in base oil. In other words, Fusing point of urea compound of the configuration employing to the present invention is equal to or less than 230 degrees Celsius. In addition, Because it is difficult for carbon number of R3 to obtain monoamine of the long chain that compound of greater than 18 becomes charge stock in economic price, it is not suitable for practical use. R2 is carbohydrate moiety of the diisocyanate which is charge stock, and the diisocyanate which can be used to the present invention is four kinds of next. % by weight is desirable, and, in urea grease composition of 2,4- 2,6- 4,4 3,3 4,4 tolylene diisocyanate tolylene diisocyanate □CE - diphenyl-methane diisocyanate □CE - dimethyl - □CE - biphenylene diisocyanate present invention, content 2-30 of the diurea compound which is *zo** agent are 5-25 % by weight. When content of diurea compound is less than double quantity %, there is a little *zo** effect, and should not grease-shaped, in addition, grease becomes too hard, and enough lubrication effect is not provided when 30 % by weight is exceeded. Base oil to use to the present invention is lube oil known to the public, and it is liquid petrolatum, alpha - olefin oligomer, silicone oil, diester oil, triester oil, tetraester oil, fluorine oil, phosphate oil, castor oil, phenyl ether oil, alkyl naphthalene, alkylene glycol. Various additive such as oxidation inhibitor, anti-rust agent, extreme pressure agent can be added to urea grease of the present invention separately from component of original without harming the property to be directed to, even more particularly, to improve the ability. In addition, Preferably, in the present invention, as for the velocity cooling solution becoming urea compound than base oil to room temperature, 10 degrees Celsius per second are older than more than 5 degrees Celsius per second. If it is more than 5 degrees Celsius per second, cooling velocity varies on the way, and it does not interfere either. When it cooled off at the rate of under 5 degrees Celsius per second, there is easy to be aggregate more than 20 μ m and is not suitable for the present invention. If chiller to

use to the present invention is the apparatus that desired cooling velocity is provided, is not limited in particular. By way of example only, Desired cooling velocity is provided by the method which solution is poured into on the stainless board which service water is sprayed from a bottom, and cool off serially. Urea grease prevents flocculation between special crystal by passing the temperature which formula of grease used for the present invention cools molecular of the Urea chemical agent which dispersed within base oil in high temperature separately in rapid, and crystallize quickly. Because when solution was cooled slowly, in the vicinity of the temperature that urea compound crystallizes, condition or the latter that urea chemical agent dissolved in urea chemical agent and the base oil which crystallized coexists are easy to crystallize in form getting twisted up in the urea chemical agent which crystallized in a point, it is thought that the aggregate which is giant generates, this can be prevented by cooling the whole grease uniformly rapidly. Formula of the present invention is to do the low noise urea grease which dissolution quench does mixture becoming low melting urea compound than base oil, and is provided in grease keeping the gel structure which is stable in high temperature. Of course, it is equal to or less than 230 degrees Celsius, and the grease which assumed low melting urea compound z_o^* agent does drip in drip-point check (JIS.K 2220 5.4). In addition, When temperature is put up little by little, it is more than 160 degrees Celsius, and most become liquid, and it can employ only in temperature of less than or equal to 150 degrees Celsius for reality. As a result of having repeated investigation for the purpose of scholars of present invention improving this point, that drip-point becomes high by means of mixing grease composition provided by formula of grease used for the present invention more than two kinds is found, even if it employs in the high temperature which 150 degrees Celsius to be directed to are older than, having the gel structure which is stable, if it is completed in noise urea grease low, it was reached. In other words, Drip-point is high, and tei^* sound urea grease having the gel structure which is stable in high temperature is provided by mixing higher than two kinds of foreign things of urea chemical agent at the rate of option among the low noise urea grease which dissolution quench does the composition which fusing point suffers from than low melting urea compound of less than or equal to 230 degrees Celsius and base oil, and is provided. Rise of drip-point is recognized in the mix proportion that two kinds of weight ratios of z_o^* agent of grease mixed for mix proportion of grease seem to become 1 to 99, but, the mix proportion that 5-95 pairs weight ratios of foreign z_o^* agent seem to become 95-5 is desirable so that 250 degrees Celsius get grease having drip-point older than. As thus described it is conceivable when each urea compound reinforces gel structure as for the reason why drip-point becomes high by means of mixing mutually, but, it is not reached till theoretical is elucidated. [embodiment] the present invention is explained by the following embodiment, a comparative example concretely. As for the abbreviation, the language, the check method to use in embodiment, a comparative example here, following. (1) TDI2,4 - tolylene diisocyanate (2) MDI4,4 $\square\text{C}\text{E}$ - diphenylmethane diisocyanate (3) 4,4 TODI3,3 $\square\text{C}\text{E}$ - dimethyl - $\square\text{C}\text{E}$ - biphenylene diisocyanate (4) Mineral oil paraffin series purification mineral oil 40 degrees Celsius kinematic viscosity 67cSt(5) Ester pentaerythritol tetraester 40 degrees Celsius kinematic viscosity 31cSt(6) Temperature when the solution which solution temperature urea compound completely dissolves in base oil is moved to cooling operation (7) Average of velocity cooling solution in average cooling temperature cooling operation to room temperature neighborhood. By way of example only, If it cools off from 200 degrees Celsius to 20 degrees Celsius in five seconds, average cooling velocity becomes 36 degrees Celsius /sec. (8) Consistency JIS K 2220 5.3 is depended on. (9) Drip-point JIS K 2220 5.4 is depended on. Number foreign element check of aggregate (JIS K 2220 5.9) is applied, grease is applied to slide glass, cover glass is covered with, and grease film thickness 30 is done in μm , it is observed in light microscope (magnification 100 times), 20 μm in within the limit of 0.46mm \times 0.64mm counts aggregate of size older than. *anderon* value *anderonmeta* rolls, and is employed as jolt measuring instrument of bearing broadly, inner race of bearing is turned, it is apparatus indicating oscillation of the outer ring which burdened thrust load in unit of *anderon*. With the present invention, Bearing is turned in bearing 6200, quantity 0.30g that grease is not allotted for, bearing inner race number of revolutions 1800r.p.m, examination condition of thrust load 2.0kg for one minute, designation value in High Band after one minute (1800-10000Hz) is read as *anderon* value. Grease 1-11 used for the present invention were got by the following production method. The solution which heat dissolved amine in in base oil of solution and the balance which it heats, and dissolved isocyanate in 1/2 base oil quantity is mixed in temperature of 70-100 degrees Celsius, and

it is reacted, while spatulating, it heats, and it is drained from a bottom into the product made in stainless vessel which city water continues being sprayed on, and the solution which is uniformity and the event that it was are crowded with, and it is cooled, after getting cold to room temperature neighborhood, three roll mill was passed twice. Temperature change in cooling connects thermocouple to mounting, recorder in container made by stainless, and it measures, the mean cooling velocity calculated from this temperature change log. Drip-point of grease 1-11 is the same level as commercial lithium soap base grease, but, there is not quite aggregate more than 20 μm , and *anderon* value is 11.0-14.0, and what marketing urea grease shown in comparative example 8-11, marketing low noise ability low more superior than noise lithium soap base grease comprise is clear. It was produced in blending same as grease 7, and grease 12,13 changed average cooling velocity. Average cooling velocity 13 degrees Celsius /sec, grease 13 is average cooling velocity 153 degrees Celsius /sec, and it is compared with grease 7 of average cooling velocity 54 degrees Celsius /sec, and grease 12 approximately comprises low noise ability of the dimension. Therefore, Average cooling velocity is changed in this area, and low noise ability understands that there is not effect. It is the grease which a weight ratio of zo^* agent seems to suffer at 99:1, and example 1 mixes grease 1 and grease 4, and three roll mill is passed twice, and was got. This grease was drip-point 230 degrees Celsius, number 0 of aggregate more than 20 μm , *anderon* value 12.5. It is the grease which a weight ratio of zo^* agent seems to suffer at 95:5, and grease 1 and grease 4 are mixed, and example 2 passes three roll mill twice, and was got. This grease was drip-point 254 degrees Celsius, number 0 of aggregate more than 20 μm , *anderon* value 12.5. It is the grease which a weight ratio of zo^* agent seems to suffer at 65:35, and grease 1 and grease 4 are mixed, and example 3 passes three roll mill twice, and was got. This grease was drip-point 272 degrees Celsius, number 0 of aggregate more than 20 μm , *anderon* value 12.5. Embodiment 4, grease 1 and grease 4 and grease 2, weight ratio 37.7: of zo^* agent 28.3: It is the grease which it is mixed to become 34.0, and three roll mill is passed twice, and was got. This grease was drip-point 281 degrees Celsius, number 0 of aggregate more than 20 μm , *anderon* value 12.5. Because example 2,3,4 is high drip-point, even temperature more than 150 degrees Celsius that cannot employ can employ in lithium soap base grease and marketing urea grease, marketing are extremely useful grease having low noise ability superior to noise lithium soap base grease low. As for comparative example 1,2, carbon number 7 of R1,R3 of urea chemical agent generated by reaction with diisocyanate is equal to or less than to use n-butylamine, n-heptyl amine respectively. Because this grease provided by production method the same as grease 1-11 is 230 degrees Celsius both, and zo^* agent does not dissolve in base oil, even if quench is done at the rate of higher than 5 degrees Celsius /sec, there is the aggregate that 20 μm is older than, and *anderon* value is each 38.0, 27.0 and big value. Comparative example 3,4 contain cycloalkyl group or phenyl group in R1,R3 of urea chemical agent generated by reaction with diisocyanate to use cyclohexylamine, p-phenetidine respectively. Because this grease provided by production method the same as grease 1-11 is 230 degrees Celsius both, and zo^* agent does not dissolve in base oil, even if quench is done at the rate of higher than 5 degrees Celsius /sec, there is the aggregate that 20 μm is older than, *anderon* value is each 26.0, 46.0 and big value. After comparative example 5 was blending same as grease 2, and having reacted, heat agitation is left at 170 degrees Celsius, it is grease provided by production method to pass three roll mill after cooling at the rate of 56 degrees Celsius /sec twice. Because it did not make base oil completely dissolve this grease urea compound, there is aggregate more than 20 μm , *anderon* value is 33.0 and big value, too. Comparative example 6,7 react in blending same as grease 2, heat is spatulated, after having made base oil dissolve urea chemical agent in complete at 223 degrees Celsius, it is cooled in each mean cooling velocity 2.4 degrees Celsius /sec, 4.1 degrees Celsius /sec, it is grease provided by production method to pass three roll mill twice. Because cooling velocity to average this grease is less than 5 degrees Celsius /sec, there is aggregate more than 20 μm , *anderon* value is massive with 24.0 both. Urea grease that comparative example 8,9 market, comparative example 10,11 are marketing low noise lithium soap base grease. The result is made table, and it is given next.

	グリース 1	グリース 2	グリース 3	グリース 4	グリース 5	グリース
アミン [重量%]	オクタデシルアミン 4.83	ドデシルアミン 6.88	テトラデシルアミン 7.29	オクタデシルアミン 6.61	3,5,5-トリメチルヘキシルアミン 6.24	ドデシルアミン 7.16
イソシアネート [重量%]	TDI 5.17	同左 5.12	同左 4.71	同左 3.39	同左 5.76	MDI 4.84
基油 [重量%]	鉱油 90.0	同左 88.0	同左 88.0	同左 90.0	同左 88.0	同左 88.0
溶液温度	228°C	223°C	220°C	198°C	227°C	226°C
平均冷却速度	94°C/sec	64°C/sec	61°C/sec	53°C/sec	79°C/sec	62°C/sec
稠度	215	210	235	240	238	244
滴点	223°C	220°C	209°C	182°C	217°C	221°C
20 μm以上の凝集体個数(0.46mm×0.64mm×30 μmあたり)	0	0	0	0	0	0
アンデロン値	12.5	12.5	11.0	12.5	14.0	12.0

	グリース 7	グリース 8	グリース 9	グリース 10	グリース 11	グリース 12	グリース
アミン [重量%]	オクタデシルアミン 8.14	ドデシルアミン 10.21	同左 6.88	同左 7.16	同左 10.21	オクタデシルアミン 8.14	同左 8.14
イソシアネート [重量%]	MDI 3.86	TDI 4.79	TDI 5.12	MDI 4.84	TDI 4.79	MDI 3.86	同左 3.86
基油 [重量%]	鉱油 88.0	同左 85.0	エステル 88.0	同左 88.0	同左 88.0	鉱油 88.0	同左 88.0
溶液温度	203°C	225°C	223°C	225°C	225°C	203°C	203°C
平均冷却速度	54°C/sec	80°C/sec	64°C/sec	62°C/sec	80°C/sec	13°C/sec	153°C/sec
稠度	238	245	237	250	261	268	214
滴点	186°C	210°C	214°C	220°C	207°C	186°C	186°C
20 μm以上の凝集体個数(0.46mm×0.64mm×30 μmあたり)	0	0	0	0	0	0	0
アンデロン値	11.0	10.5	13.0	12.5	13.0	11.5	11.5

	実施例 1		実施例 2		実施例 3		実施例 4		
アミン [重量%]	オクチルアミン 4.83	オクタデシルアミン 6.61	オクチルアミン 4.83	オクタデシルアミン 6.61	オクチルアミン 4.83	オクタデシルアミン 6.61	オクチルアミン 4.83	オクタデシルアミン 6.61	ドデシルアミン 6.88
イソシアネート [重量%]	TODI 5.17	TODI 3.39	TODI 5.17	TODI 3.39	TODI 5.17	TODI 3.39	TODI 5.17	TODI 3.39	TODI 5.12
基油 [重量%]	鉱油 90.0	鉱油 90.0	鉱油 90.0	鉱油 90.0	鉱油 90.0	鉱油 90.0	鉱油 90.0	鉱油 90.0	鉱油 88.0
溶液温度	228°C	198°C	228°C	198°C	228°C	198°C	228°C	198°C	223°C
平均冷却速度	94°C/sec	53°C/sec	94°C/sec	53°C/sec	94°C/sec	53°C/sec	94°C/sec	53°C/sec	64°C/sec
グリース混合比	99 : 1		95 : 5		65 : 35		40 : 30 : 30		

	実施例 1	実施例 2	実施例 3	実施例 4
増稠剤重量比	99 : 1	95 : 5	65 : 35	37.7 : 28.3 : 34.0
稠度	217	220	228	223
滴点	230°C	254°C	272°C	281°C
20 μm以上の凝集体個数(0.46mm×0.64mm×30 μmあたり)	0	0	0	0
アンデロン値	12.5	12.5	12.5	12.5

	比較例 1	比較例 2	比較例 3	比較例 4	比較例 5	比較例 6	比較例 7
アミン [重量%]	n-ブチルアミン 3.56	n-ヘプチルアミン 4.65	シクロヘキシルアミン 6.39	p-フェニチジン 7.64	ドデシルアミン 6.88	同左	同左
イソシアネート [重量%]	TODI 6.44	同左 5.35	同左 8.61	同左 7.36	同左 5.12	同左	同左
基油 [重量%]	鉱油 90.0	同左 90.0	同左 85.0	同左 85.0	同左 88.0	同左 88.0	同左 88.0
溶液温度	230°C溶解せず	同左	同左	同左	170°C溶解せず	223°C	同左
平均冷却速度	82°C/sec	同左	同左	同左	56°C/sec	2.4°C/sec	4.1°C/sec
稠度	273	288	263	275	302	283	280
滴点	275°C	271°C	285°C	288°C	220°C	220°C	220°C
20 μm以上の凝集体個数(0.46mm×0.64mm×30 μmあたり)	14	9	10	18	12	7	5
アンデロン値	38.0	27.0	26.0	46.0	33.0	24.0	24.0

	比較例 8	比較例 9	比較例 10	比較例 11
	市販ウレアグリース	市販ウレアグリース	市販低騒音リチウム石鹼グリース	市販低騒音リチウム石鹼グリース
稠度	267	271	235	268
滴点	285°C	260°C	192°C	194°C
20 μm 以上の凝集体個数 (0.46mm \times 0.64mm \times 30 μm あたり)	17	12	0	4
アンデロン値	39.0	35.0	18.0	21.0

[an effect of the invention] there is not the aggregate that 20 μm of the diurea chemical agent corpuscule which is the zo* agent is older than without losing the high temperature stability which is feature of urea grease by formula of low noise urea grease composition concerning the present invention, minute diurea chemical agent crystal of less than or equal to 15 corpuscule length of a particle (or, fiber length) μm getting the urea grease composition which dispersed in uniformity was succeeded in. Low noise ability provided low noise urea grease composition comprises high heat resistance than a case of one kind of grease to mix grease comprising of foreign urea compound more than two kinds and to be superior to marketing low noise lithium soap base grease and a thing having high temperature stability are ensured. In other words, Formula of low noise urea grease composition concerning the present invention comprises heat resistance, superior effect to be able to provide noise urea grease low of durability than conventional low noise lithium soap base grease.

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